IS 3928 (1986): Styralyl Acetate [PCD 18: Natural and Synthetic Fragrance Materials]

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IS : 3928 - 1986
(Reaffirmed 2000)

Indian Standard

SPECIFICATION FOR STYRALYL ACETATE

(First Revision)

UDC 665·57:547·568·1·292

BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Gr 3

April 1987

Indian Standard

SPECIFICATION FOR STYRALYL ACETATE

(First Revision)

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Indian Standard

SPECIFICATION FOR STYRALYL ACETATE

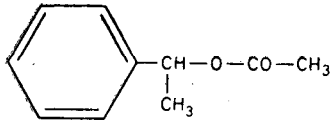
(First Revision)

0. FOREWORD

0.1 This Indian Standard (First Revision) was adopted by the Indian Standards Institution on 15 September 1986, after the draft finalized by the Natural and Synthetic Perfumery Materials Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

0.2 This Indian Standard was first published in 1966. The Sectional Committee responsible for its preparation felt that it should be revised with a view to bring it in line with trade practices in perfumery technology and also to align with the quality of the material currently manufactured and sold in the country.

0.3 Styralyl acetate ($C_{10}H_{12}O_2$) is found in the oil of gardenia flower from *Gardenia* spp fam. Rubiaceae. It is a commercially available perfumery chemical used extensively in gardenia scents and to some extent in tuberose, jasmine and other floral compositions. It has the following structural formula:



Styralyl acetate (molecular mass 164·21)

0.4 A new requirement of styralyl acetate, percent by mass minimum along with gas chromatographic analysis for determination of styralyl acetate has been incorporated in this revision based on data generated through indigenous testing. Requirements of relative density and refractive index have also been modified.

0.5 In the preparation of this standard, considerable assistance has been derived from the Givaudan Index (Second Edition), 1961, published by Givaudan-Delawanna Inc, New York.

0.6 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for styralyl acetate.

2. TERMINOLOGY

2.1 For the purpose of this standard, the definitions given in IS : 6597-1972† shall apply.

3. REQUIREMENTS

3.1 Description

3.1.1 The material shall be obtained by esterification of phenylmethyl carbinol with acetic acid.

3.1.2 The material shall be a colourless liquid, free from sediment, suspended matter and adulterants.

3.1.3 The material shall be examined for its colour, clarity, suspended matter and sediment as prescribed in IS : 326 (Part 2)-1980‡.

3.2 Solubility — The material shall be soluble in 5 volumes of ethyl alcohol (60 percent by volume), when tested as prescribed in IS : 326 (Part 6)-1986§.

*Rules for rounding off numerical values (*revised*).

†Glossary of terms relating to natural and synthetic perfumery materials.

‡Methods of sampling and test for natural and synthetic perfumery materials: Part 2 Preliminary examination of perfumery materials and samples (*second revision*).

§Methods of sampling and test for natural and synthetic perfumery materials: Part 6 Determination of solubility in ethanol (*second revision*).

3.3 The material shall also be tested olfactorily and specially for by-notes as prescribed under **4** and **5** of IS : 2284-1963*.

3.4 The material shall also comply with the requirements given in Table 1.

TABLE 1 REQUIREMENTS FOR STYRALYL ACETATE

Sl No.	Characteristics	Requirement	Method of Test, Ref to Cl No. in	
			Indian Standard	Appendix
(1)	(2)	(3)	(4)	(5)
i)	Odour	An intense green odour suggestive of gardenia	IS : 2284-1963*	
ii)	Relative density at 27°C (*see* Note 1)	1·018 to 1·021	IS : 326 (Part 3)-1980†	
iii)	Refractive index at 27°C (*see* Note 2)	1·488 to 1·491	IS : 326 (Part 5)-1986†	
iv)	Acid value, *Max*	1·0	IS : 326 (Part 7)-1980†	
v)	Styralyl acetate, percent by mass, *Min*	98		A

NOTE 1 — The correction factor for specific gravity for each degree centigrade change in temperature is *0·000 64 [5.2 of IS : 326 (Part 3)-1980†]*.

NOTE 2 — The correction factor for refractive index for each degree centigrade change in temperature is 0·000 38 [IS : 326 (Part 5)-1986†].

*Method for olfactory assessment of natural and synthetic perfumery materials.

†Methods of sampling and test for natural and synthetic perfumery materials:
Part 3 Relative density (*second revision*).
Part 5 Determination of refractive index.
Part 7 Determination of acid value (*second revision*).

4. PACKING AND MARKING

4.1 The material shall be supplied in glass bottles, or in any suitable container as agreed to between the purchaser and the supplier.

4.2 The containers may also be marked with the Standard Mark.

NOTE — The use of the Standard Mark is governed by the provisions of the Bureau of Indian Standard Act 1986 and the Rules and Regulations made thereunder. The Standard Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well defined system of inspection, testing and quality control which is devised and supervised by BIS and operated by the producer. Standard marked products are also continuously checked by BIS for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

*Method for olfactory assessment of natural and synthetic perfumery materials.

5. SAMPLING

5.1 Representative samples of the material shall be drawn as prescribed in IS : 326 (Part 1)-1984*.

6. TEST METHODS

6.1 Tests shall be conducted as prescribed under **3.1, 3.2, 3.3** and the appropriate references specified in col 4 and 5 of Table 1.

6.2 Quality of Reagents — Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1977†) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

APPENDIX A

[*Table* 1, *item* (v)]

GAS CHROMATOGRAPHIC ANALYSIS FOR DETERMINATION OF STYRALYL ACETATE

A-0. GENERAL

A-0.1 The chromatographic conditions given here are for guidance only.

A-0.2 Outline of the Method — A sample of the material is injected into the gas chromatograph where it is carried by the carrier gas from one end of the column to the other. During its movement, constituents of the sample undergo distribution at different rates and ultimately get separated from one another. The separated constituents emerge from the end of the column one after another and are detected by suitable means whose response is related to the amount of a specific component leaving the column.

*Methods of sampling and test for natural and synthetic perfumery materials: Part 1 Sampling (*second revision*).

†Specification for water for general laboratory use (*second revision*).

A-1. APPARATUS

A-1.1 Any gas chromatograph capable of being operated under conditions suitable for resolving the individual constituents into distinct peaks may be used. The typical chromatograph for styralyl acetate using a chromatograph with the following chromatographic conditions is shown in Fig. 1.

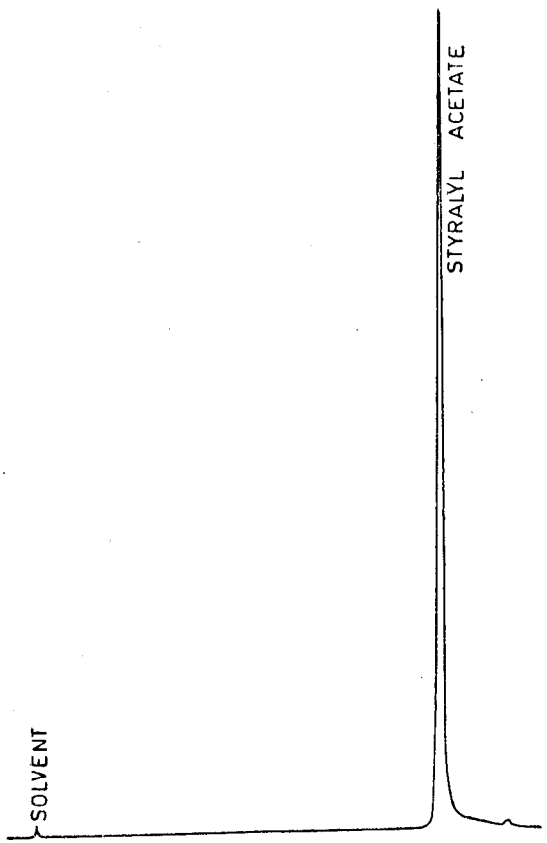


FIG. 1 TYPICAL CHROMATOGRAM OF STYRALYL ACETATE

a) *Sample*: Styralyl acetate

1) Material	Copper
2) Length	12 m
3) Outer diameter (OD)	0·635 cm
4) Inner diameter (ID)	0·476 cm
5) Stationary phase	FFAP*, 10 percent by mass
6) Solid support	Chromasorb WAW 60-80 mesh

b) *Carrier Gas*: Nitrogen

c) *Conditions*

1) Column temperature isothermal	180°C
2) Injection port temperature	200°C
3) Carrier gas flow	20 ml/min
4) Inlet pressure	3·5 kg/cm^2

d) *Detector*

1) Type	F.I.D
2) Temperature	280°C

e) *Recorder*

1) Span	1 mV
2) Chart speed	0·25 cm/min

f) *Attenuation*: 32

NOTE — This analysis may also be accomplished with columns containing carbowax-20 M, DE. G. S (Diethylene Glycol Succinate).

A-2. PROCEDURE

A-2.1 Conduct the flow of the carrier gas and inject the sample at inject port where it is vaporized and well-mixed with the carrier. This is led into the chromatographic column wherein vaporized constituents of the sample are separated out by virtue of their differing interaction with the stationary phase. As the different constituents pass through the detector, they give signals corresponding to the amount of particular constituent leaving the column. The detector signal, on transmission to the recorder, plots the chart. From the specific area under various peaks corresponding to specific constituents, the quantities of different constituents are determined.

NOTE — For the separation to be efficient, it is necessary that the column is maintained at the temperature suggested throughout the time required for resolution of the constituents.

*Free fatty acid phase (FFAP) is carbowax-20M treated with nitrophthalic acid.

A-3. CALCULATION

A-3.1 Area Measurement (*see* **Note 1**) — Since normal peaks approximate a triangle, the area is measured by multiplying the peak height with the width of half-height. The normal peak base is not taken since large deviations may be observed due to tailing or adsorption. This technique is rapid, simple and fairly accurate when peaks are symmetrical and are of reasonable width.

A-3.2 Area Normalization (*see* **Note 2**) — By normalization, it is meant, calculating the percentage composition by measuring the area of each and dividing the individual areas by total area, for example:

$$\text{Percentage of } A = \frac{\text{Area of } A}{\text{Total area}} \times 100$$

NOTE 1 —Other methods of area measurement, namely, triangulation disc integrator and electronic digital integrator, if fixed with GLC machine, would be of great advantage.

NOTE 2 — Internal standardization can be used if pure appropriate internal standard is available. This method is known as relative or indirect calibration.

(*Continued from page* 2)

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[*Page* 5, *Table* 1, *Sl No.* (v), *col* 3] — Substitute '99' *for* '98'.

(PCD 18)

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